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During the first two years of this program, significant effort was concentrated on securing a source and establishing processing routes for the materials under consideration. This was necessarily accompanied by the development and use of scoping techniques for initial characterization of the candidate materials and processes. Such techniques need to be rapid and informative tests which are forgiving of sample imperfections, so that a few parameters of interest of a large number of sample materials can be easily compared. In this study the techniques based upon high temperature microindentation were chosen to rank the various static and time-dependent mechanical properties, and scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) was used for microstructural and chemical evaluation.

During the third program year, the study of the four transition metal beryllides was continued and expanded to include high temperature compression testing. Given the very limited quantity of material available, the range of testing was limited. Small cylinders, (2.5 mm. diameter by 5 mm. long) were electro-discharge machined from the sample material and were tested at high temperature using a servo-hydraulic testing frame. This effort expanded upon the second year's microhardness and microindentation creep results by the first known effort to examine these materials using uniaxial compression testing.

Developing and Understanding the High Temperature Behavior of Niobium Aluminides and Niobium Aluminide Composites

Final Technical Report

Submitted to

Air Force Office of Scientific Research

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Austin, TX 78712

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Introduction

The present three year study has sought to understand the potential for, and limitations to, development of high temperature structural materials based on niobium aluminide and transition metal beryllide intermetallic compounds. Beginning with a broad examination of some important properties, this project has identified promising areas for research and culminates with significant additions to the body of knowledge on both of these classes of materials.

The future of aeropropulsion systems requires structural materials with ever increasing temperature capabilities coupled with low density and oxidation resistance. Intermetallic compounds have been widely studied as candidate replacement systems for the nickel base superalloys. Historically, the most widely studied intermetallic compounds have been the nickel, iron and titanium aluminides. However, these systems are limited by a service ceiling of about 1000°C due to their inadequate strength and creep resistance at higher temperatures. Properties such as high strength, structural stability and creep resistance, are known to scale with melting temperatures. Consequently, further development demands expanding our horizons to include higher melting temperature intermetallic compounds that satisfy all of the other requirements of high temperature structural applications, such as low density, corrosion resistance and ease of processing.

This problem can be approached from a number of directions. The widely studied aluminides have attempted to surpass superalloys by exploiting the Ni_3Al reinforcing phase in monolithic form. The progression to the other systems was a natural step in terms of increased

temperature capability and lower density. To extend this incremental progress to the limits of current processing technology, one needs to study the niobium aluminum system. These compounds, $NbAl_3$, Nb_2Al and Nb_3Al, indeed represent the most advanced systems which can be produced using current melting technology for aerospace grade materials. An alternate approach towards advances in high temperature materials leads to interest in the compounds of beryllium. Beryllide compounds such as Ta_2Be_{17}, Nb_2Be_{17}, $TiBe_{12}$, and $ZrBe_{13}$ possess the unique advantage of

Table I

Intermetallic Compound	**CTE (10^{-6} / °C)**
FeAl	21.0
Ni_3Al	16.0
NiAl	16.0
TiAl	10.0
Ti_3Al	10.0
$TiBe_{12}$	16.0
$ZrBe_{13}$	16.0
Nb_2Be_{17}	15.3
Ta_2Be_{17}	16.0

extremely low densities. This most important aerospace design criteria, coupled with high melting temperature and high stiffness, warrants further investigation of these materials. Furthermore, excellent parity with the coefficient of thermal expansion of many potential composite matrices demands consideration as a reinforcement in metal or intermetallic matrix composites (Table I).

To compare the three niobium aluminides and four beryllides with the more familiar structural aluminides, Table II lists these systems in terms of melting temperature, density, and crystal structure. It should be noted that the niobium aluminides melt in the range of 1605°C - 1960°C, and the beryllides in the range of 1570°C - 1980°C, surpassing nearly all of the common aluminides. Table II also demonstrates the clear superiority of the beryllides over their corrivals in terms of density. Also evident is

Table II

Intermetallic Compound	**T_m (°C)**	**Crystal Structure**	**Density (g/cm^3)**
FeAl	1340	B2	6.0
Ni_3Al	1390	$L1_2$	7.5
TiAl	1452	$L1_0$	3.9
Ti_3Al	1472	DO_{19}	4.2
$TiBe_{12}$	**1570**	**$D2_{0b}$**	**2.3**
$NbAl_3$	**1605**	**DO_{22}**	**4.6**
NiAl	1638	B2	5.8
$ZrBe_{13}$	**1645**	**$D2_3$**	**2.7**
Nb_2Be_{17}	**1800**	**hR19**	**3.3**
Nb_2Al	**1870**	**$D8_b$**	**6.9**
Nb_3Al	**1960**	**A15**	**7.3**
Ta_2Be_{17}	**1980**	**hR19**	**4.2**

the complexity of the crystal structures of the subject systems. The intricate crystal structures support few dislocation slip systems, thereby affecting deformation mechanisms through numerous locking mechanisms, predominance of non-dislocation modes, and high cross slip related energies. As a consequence of these slip difficulties, extraordinary high temperature strengths and creep resistances are also expected.

Work on these systems to date is sparse. Although beryllides were examined as structural compounds to some degree in the 1960's [1-4], these efforts were abandoned due to the lack of low temperature ductility. Recently, there has been a renewed interest in these compounds. Researchers at Battelle Pacific Northwest Laboratory have been extremely active on the mechanical properties of the Be-Nb system. Bruemmer and co-workers examined strength and toughness of vacuum hot pressed $NbBe_{12}$ by four point bend testing and microhardness. They observed stacking faults on {110} and {100} planes. Perfect dislocations were also found above 1000°C [5,6]. Charlot and co-workers have examined sputter deposited $NbBe_{12}$ and Nb_2Be_{17} by microhardness [7]. Nieh et. al. at Lockheed have also examined vacuum hot pressed Be-Nb compounds as well as $TaBe_{12}$, $ZrBe_{13}$ and Ni-Be compounds by hot indentation tests [8]. A recent paper by the same workers [9] examined the mechanical properties of NiBe at ambient and elevated temperatures. They found that at high temperatures, NiBe exhibits up to 30 % ductility.

The niobium aluminum phase diagram was first assessed in the middle 1960's [10] and has been widely studied because of the superconducting properties of niobium and of the A15 Nb_3Al compound. In 1975, Shechtman and Jacobson began the modern interest in the niobium aluminides as structural materials [11]. Their study of room temperature deformed $NbAl_3$ yielded important data on deformation modes in these compounds. The observation of [110] dislocations and unidentified dislocation loops on (100) planes was important in understanding the limited ductility. These defects were reported to be energetically unfavorable and to require a high critical resolved shear stress for motion, making glide difficult. Furthermore, Shechtman and Jacobson identified

twinning modes which, although difficult to generate, may account for the limited plasticity that they observed.

More recently, the majority of work on the niobium aluminides has been concerned with processing and phase stability issues, including oxidation behavior. Many workers have thoroughly investigated the powder processing methods that are applicable to this and similar systems. [12-14] Another widely studied processing route in recent years is rapid solidification from the melt. Originally developed as advantageous for production of superconducting Nb_3Al, rapid solidification may also result in retained supersaturated solid solution [15-18] which may be useful for ductilization. [19] High temperature service of any material may well be limited by oxidation concerns. Accordingly, many workers have considered the inherent corrosion resistance of these aluminides and schemes for enhancing it. Hebsur et.al. have found chromium additions of about 8 weight percent to be particularly effective. [20,21] Perkins, Chiang, et. al. have likewise assessed the kinetics of scale formation in a wide range of binary and ternary alloys based on this system. [22,23] Perhaps the most complete study and accumulation of oxidation data is that of Meier and Thompson for DARPA. [24]

Mechanical property data is only recently beginning to surface. Anton and Shah have been studying the niobium aluminides along with other intermetallic compounds for some time [19, 25-27]. Stoloff has compared these compounds to other refractory metal aluminides via elevated hardness and compression testing [28]. Initial results suggest that the Nb_xAl_y intermetallics hold great promise. Recently, a large number of Japanese research groups have begun investigating the niobium-aluminum compounds. Fujiwara, Yasuda and Kodama are combining mechanical

property studies w'th oxidation tests and have surmised that chromium additions are particularly beneficial towards both ends.[29].

Given these causes for interest, this research program set out to assess the future of the niobium-aluminum and transition metal-beryllium intermetallics for high temperature structural use. The primary interest was to identify promising directions for future development of these materials either in monolithic or composite form. Furthermore, an understanding of the scientific and technological issues involved in their used was sought.

First and Second Year Accomplishments

During the first two years of this program, significant effort was concentrated on securing a source and establishing processing routes for the materials under consideration. This was necessarily accompanied by the development and use of scoping techniques for initial characterization of the candidate materials and processes. Such techniques need to be rapid and informative tests which are forgiving of sample imperfections, so that a few parameters of interest of a large number of sample materials can be easily compared. In this study the techniques based upon high temperature microindentation were chosen to rank the various static and time-dependent mechanical properties, and scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) was used for microstructural and chemical evaluation.

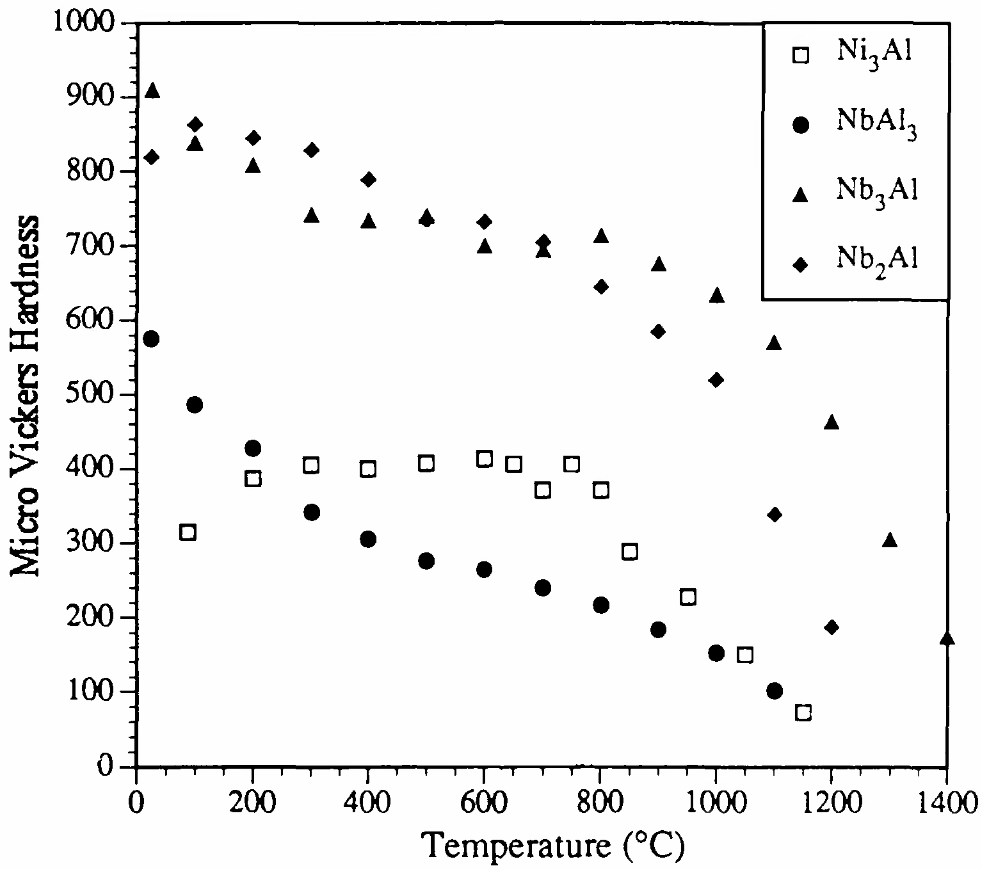
During the initial phases of this study, small quantities of the candidate aluminides and beryllides were obtained free of charge. The Nippon Mining Company, Ltd. agreed to develop, along with the University of Texas researchers, appropriate melting techniques for the production of niobium aluminides and to donate the material for this project. The various beryllium compounds were supplied by NASA-Lewis Research Center under a previous grant. The materials available from these sources was sufficient for the broad experimental work in the early program years.

During the first program year, the $NbAl_3$ sample material obtained from Nippon Mining Co. was examined for microstructural and chemical suitability and basic physical and mechanical testing was performed. During this time it was demonstrated that although microhardness testing

would be a valuable technique, the processing of these alloys would be a significant challenge. Cast niobium-aluminum alloys, using a variety of melting techniques, proved to have a high degree of microcracking and considerable chemical inhomogeneity and variation between melts.

During the second program year, all three of the niobium aluminide intermetallics were provided by the Nippon Mining Co. and the transition metal beryllide portion of this work was initiated. While the aluminides were melted by a variety of techniques, all of the beryllides were reactively sintered elemental powders which were subsequently vacuum hot pressed. This processing route formed single phase specimens of the beryllides in a fine grained condition (ASTM 12 to ASTM 9), with significant porosity. These materials were deemed suitable for mechanical testing via high temperature microindentation.

Figures 1 and 2 show the microhardness of the aluminides and beryllides, respectively, as a function of temperature. Also shown is the microhardness of Ni_3Al, which can be taken as a benchmark for the performance of structural intermetallics. Inspection of the Figures shows that significant improvements of temperature capability at constant hardness are manifested by most of these materials. Nb_2Be_{17}, Ta_2Be_{17} and Nb_3Al offer a 600°C improvement over the peak hardness of Ni_3Al, while Nb_2Al and $ZrBe_{13}$ offer an improvement of about 400°C. A more significant measure of the superiority of the beryllides over more widely studied intermetallics is shown as Figure 3. Here, density normalized values of microhardness have been plotted as a function of temperature along with data for Ni_3Al and Ti_3Al. Clearly, the specific strengths of the transition metal beryllides, as demonstrated in this manner, are far in excess of the more widely studied intermetallics.



Micro Vickers Hardness
1000
900
800
700
600
500
400
300
200
100
0
0
200
400
600
800
1000
1200
1400
Temperature (°C)
Ni3Al
NbAl3
Nb3Al
Nb2Al

Figure 1

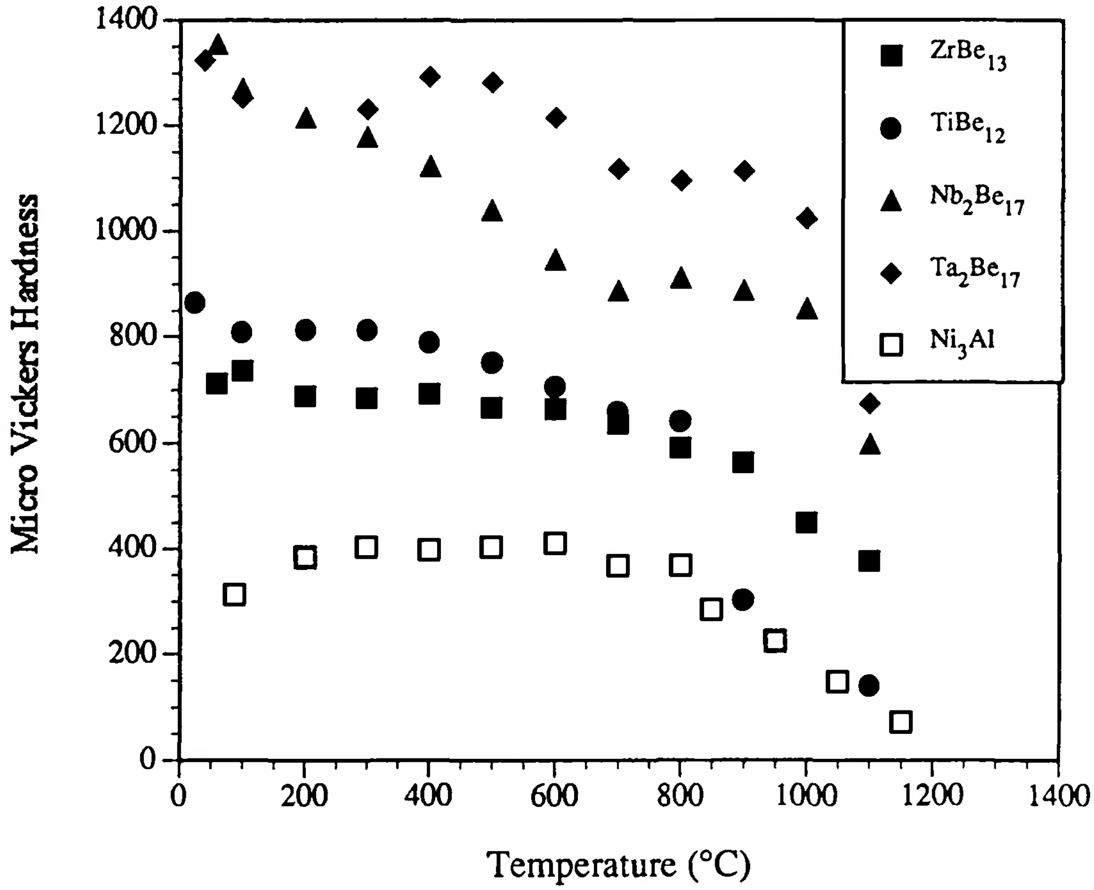
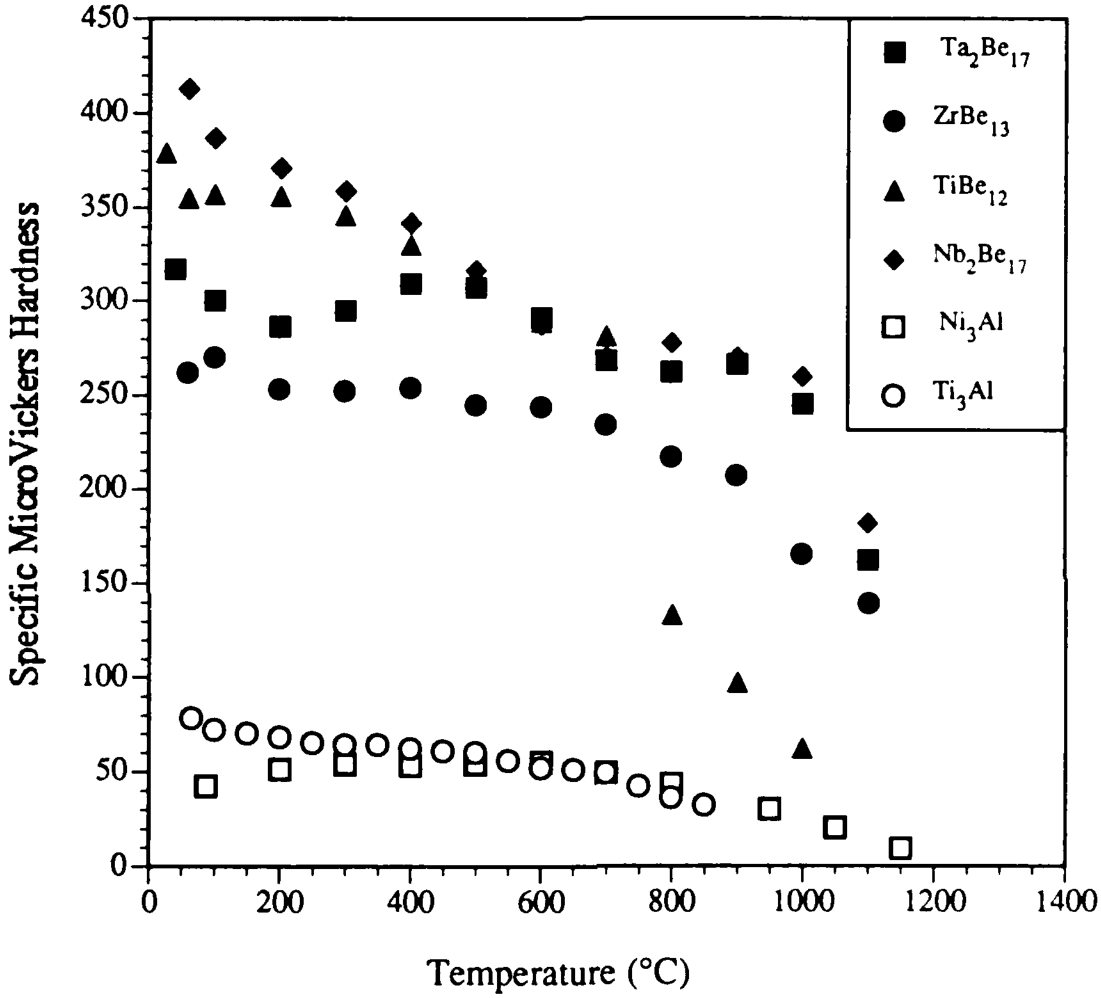

Micro Vickers Hardness
Temperature (°C)
0
200
400
600
800
1000
1200
1400
$ZrBe_{13}$
$TiBe_{12}$
Nb_2Be_{17}
Ta_2Be_{17}
Ni_3Al

Figure 2



Specific MicroVickers Hardness
Temperature (°C)
0
50
100
150
200
250
300
350
400
450
0
200
400
600
800
1000
1200
1400
Ta_2Be_{17}
$ZrBe_{13}$
$TiBe_{12}$
Nb_2Be_{17}
Ni_3Al
Ti_3Al

Figure 3

Microhardness was first used as a technique to study creep by Atkins, et. al. in 1966 [30]. By varying time on load at elevated temperatures, steady state deformation regimes can be isolated and one can extract values for steady state creep exponent (m) and activation energy (Q) from the microhardness values. Only recently has their method for simple metals been applied to and confirmed to be accurate for intermetallic compounds [8,31-33]. The temperature regime where thermally activated processes dominate can be isolated on a plot of log hardness versus inverse homologous temperature [34]. Data from the high temperature, strongly

Table III

	m (kJ/mole)	Q (kJ/mole)	Q_{sd} (kJ/mole)
Nb_3Al	4.2	531	366
Ta_2Be_{17}	4.1	317	321
$ZrBe_{13}$	4.8	115	273
Nb_2Be_{17}	5.2	442	295

temperature dependent region of such a curve is used for creep analysis By plotting the natural log of microhardness versus the natural log of hold time, a value of m can be calculated as the average linear slope. This plot is further used to determine p_0, the hardness at the time when the load is first fully applied. The hardness values are reduced by this value and its corresponding time, t_0, and then replotted to determine the activation energy, Q. The values of creep exponents and activation energies for the most promising of the systems under investigation are given in Table III. Also listed are the corresponding values for the activation energies for self diffusion for comparison.

Based on the broad scope testing of the first two years, selections for in-depth study during the final program year were made. Consideration was given not only to the results of the microhardness experiments, but also to the physical and chemical characteristics of the available material. Given the limited quantity and the sometimes poor microstructural quality of the available material, it was decided that a highly focused effort would be made on one of the niobium aluminides and all four of the transition metal beryllides during the third program year.

Third Year Accomplishments

A. Refractory Metal Beryllides.

During the third program year, the study of the four transition metal beryllides was continued and expanded to include high temperature compression testing. Given the very limited quantity of material available, the range of testing was limited. Small cylinders, (2.5 mm. diameter by 5 mm. long) were electro-discharge machined from the sample material and were tested at high temperature using a servo-hydraulic testing frame. This effort expanded upon the second year's microhardness and microindentation creep results by the first known effort to examine these materials using uniaxial compression testing.

In all cases of elevated temperature, macroscopic plasticity was demonstrated and measured in these compounds under compressive loading. The initial indications given by microindentation were followed through to this effort and, indeed, strains of up to eight percent were attained before failure. Interestingly, it was found that the cubic compound, $ZrBe_{13}$, exhibited a yield drop behavior upon initial loading at elevated temperature (1000°C - 1200°C) which was not seen on reloading. This behavior, in which the flow stress decreases sharply after initial loading, is commonly observed in impure BCC metals and has been attributed to pinning or solute drag of mobile dislocations. Since it is unlikely that such a mechanism is responsible in this case, this phenomenon clearly warrants further investigation including the study of deformation defects.

The relative strengths of Ta_2Be_{17} and Nb_2Be_{17} are also interesting results. For example, at 1200°C, the observed yield stress for the niobium

compound is more than double the 370 MPa strength of the tantalum compound. These compounds posses the same hexagonal crystal structure, melting temperatures within 180°C, similar as-tested microstructures and have very similar hardness versus temperature profiles. Despite these apparent similarities, there exists a very significant difference between the high temperature compressive behaviors of the two compounds. Some light may be shed on this seeming discrepancy by the microindentation creep values of stress exponent and activation energy listed in Table III. The relatively high values for Nb_2Be_{17} support this finding of superior high temperature strength and also demonstrates the close correspondence between time dependent microindentation and static compression in these materials.

From the results of this study, it is evident that the transition metal beryllides that we have studied have great potential as high temperature structural materials. The strongest of the four compounds, Nb_2Be_{17} maintains a strength of 525 MPa at 1200°C. $TiBe_{12}$, the lowest melting temperature compound studied, and the most comparable to the currently active alloys, was shown to far exceed presently available strength levels. Not only do these intermetallics posses high absolute strengths, but, because of extremely low density, their specific strengths far surpass any other alloy or intermetallic compound under consideration. In aero-propulsion applications, such an advantage will make even moderate melting temperature materials superior candidates.

B. Niobium Aluminides.

This portion of the program has sought to examine some of the properties related to the mechanical performance of alloys based on Nb_2Al.

Although all of the intermetallic compounds in the niobium aluminum system are being considered, in various forms, as high temperature structural materials, Nb_2Al has been shown to possess the best elevated temperature strength and creep resistance.[19,35] These qualities, combined with some attractive physical properties, make Nb_2Al and related materials promising candidates for the replacement of nickel based superalloys. Of course, there are significant limitations, such as poor oxidation resistance and low temperature brittleness, which will challenge further development. However, some workers are exploring the use of novel processing techniques, such as the directional solidification of *in-situ* composites, to exploit the benefits of the Nb_2Al compound.[36]

Three properties were selected as being fundamental to further study and development of Nb_2Al as a structural material. A characterization of the phase relationships and microstructural evolution at elevated temperature is absolutely basic. While the equilibrium binary phase diagram for niobium-aluminum alloys has been established for some time, a detailed metallographic study of microstructure, with particular attention to the compositions around Nb_2Al at high temperatures, was undertaken. In conjunction with an understanding of the microstructure, knowledge of the nanostructure, i.e. the identity and arrangement of crystal defects, is an essential basis for the design of materials systems and the explanation of observed properties. Accordingly, the second focus of this work was the identification of defects in the annealed alloys and observation of their role in macroscopic deformation. A complete characterization of the static deformation behavior of alloys near Nb_2Al using microhardness was the third effort planned for the third program year.

The four alloys that were studied here were arc melted from elemental stock and labeled R1 through R4 with increasing aluminum content. The overall compositions of the alloys spanned the known range of stability of Nb_2Al, with alloys R1 to R3 having nearly stoichiometric compositions and alloy R4 being significantly aluminum rich. A metallographic examination of the as-cast microstructures confirmed that the alloys consisted of Nb_2Al in contact with other phases. An understanding of the solidification processes and a rationale for the observed structures was straightforward and based on well known principles. A number of metastable phases had formed during casting that represented nearly all of the known phases in the system.

Upon heat treatment at 1200°C and 1350°C, the as-cast microstructures were seen to evolve and, ultimately, to reach a stable or nearly stable state. An exposure of 1350°C for 40 hours was seen to be sufficient to stabilize all of the alloys. Many of the features of the accepted phase diagram were confirmed, to the extent that the accuracy of the published data allowed. For example, 69.1 at% Nb was determined, from the microstructural evidence, to lie in the two phase $Nb_3Al + Nb_2Al$ phase field at 1200°C. Similarly, 61.7 at% Nb is beyond the solubility of aluminum in Nb_2Al at 1350°C, placing this composition in a two phase region. Both of these data would be ambiguous from the published diagram.

An entirely unexpected result was obtained from the study of microstructural evolution.[37] In all of the alloys which were aluminum rich of stoichiometry, (alloys R2 through R4), a phase developed upon heat treatment which has been unreported in the literature. In the case of the two highest aluminum content alloys, (R3 and R4), both of which contained

eutectic solid in the as-cast structure, this unknown phase appears to have transformed from the $NbAl_3$ constituent of the eutectic solid. No microstructural predecessor was evident in third aluminum rich alloy. This unexpected phase has been tentatively identified. Measurements of the composition were made by EDS and the Bravais lattice was directly hypothesized from electron diffraction data. Analysis of this information and consideration of a number of factors which are known to influence compound formation allowed an identification to be made. The new phase has been determined to be somewhat off-stoichiometric Nb_3Al_2. The measured composition is about 58 at% Nb. The hypothesized crystal structure is slightly tetragonal ($c/a < 1.1$) with a primitive unit cell containing 20 atoms per unit cell. The prototype for the conjectured structure is Zr_3Al_2.

The deformation behavior of the alloys in this study fall into two qualitative categories. The microhardnesses of alloys R1 through R3 are plotted as a function of temperature in Figure 4 and the data for alloy R4 are plotted in Figure 5. The three near-stoichiometry alloys, (R1-R3), have microhardnesses that drop slightly at low temperatures, level out to a nearly constant value through the temperature range of about 400°C to 1000°C and then rapidly fall off at high temperature. Within this category, the hardness of alloy R2, whose composition is the closest to stoichiometry, is higher at all temperatures than the hardness of the somewhat aluminum rich alloy, R3. This comparison cannot be extended to small niobium increases relative to stoichiometry because the phases in alloy R1 were too intimately mixed to allow for separate microhardness measurement of the Nb_2Al phase as in the other alloys.

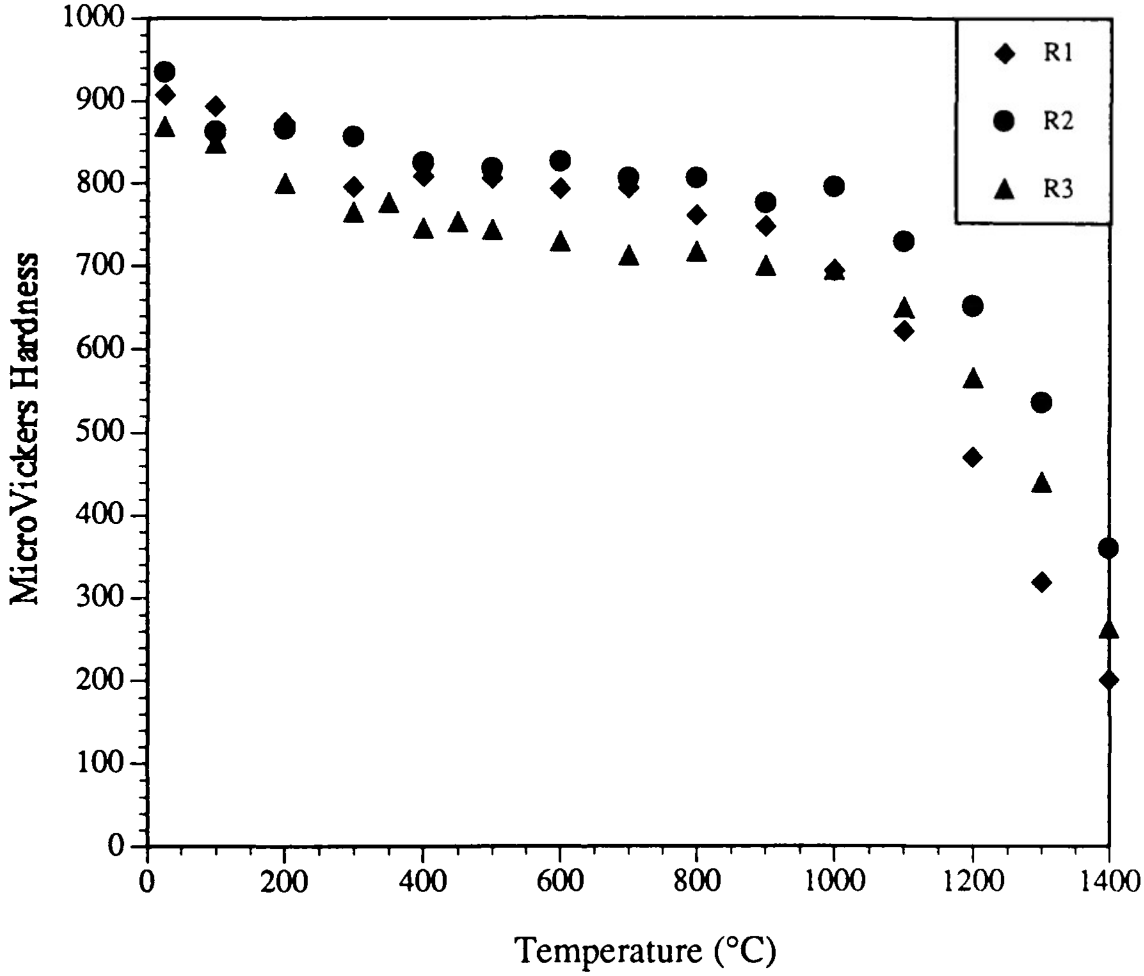
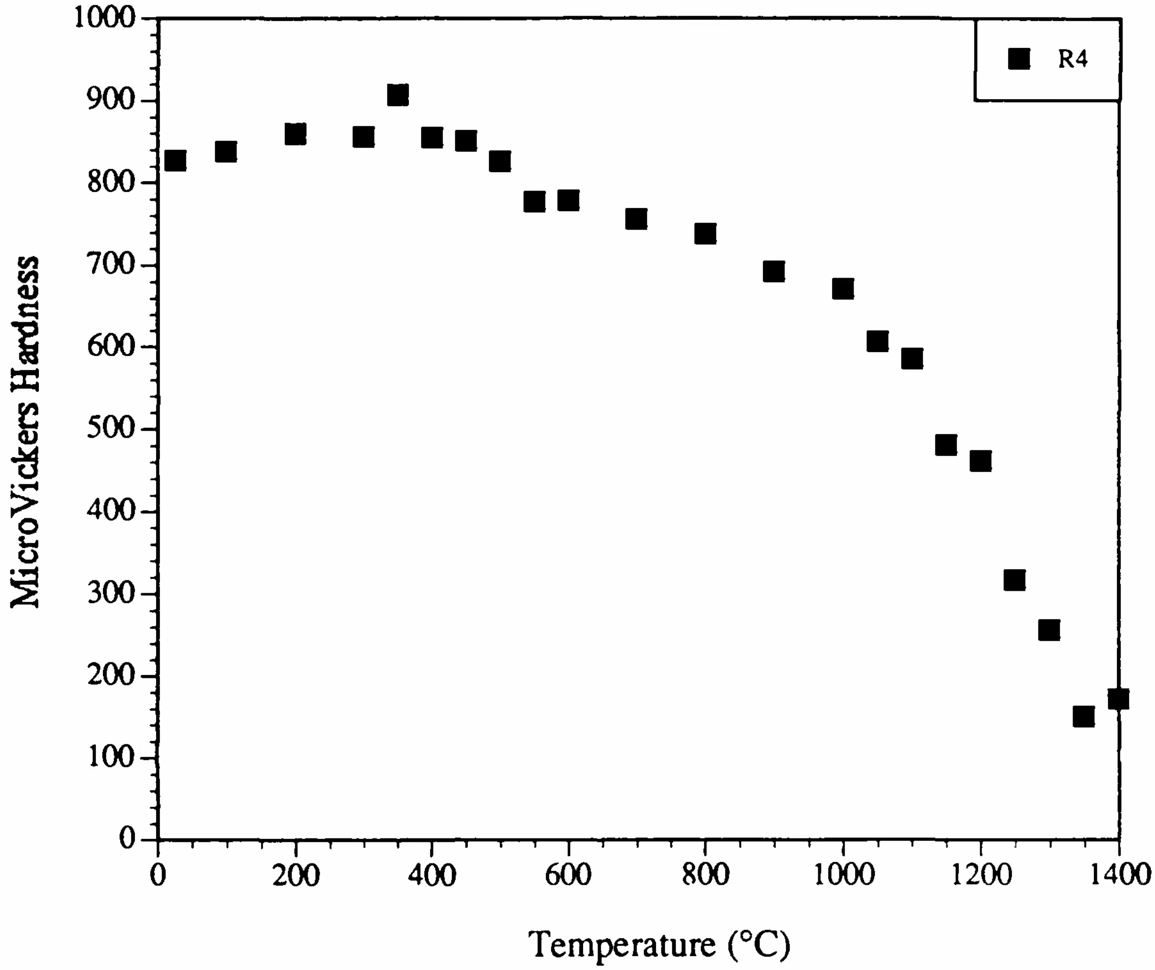

R1
R2
R3
MicroVickers Hardness
0
100
200
300
400
500
600
700
800
900
1000
0
200
400
600
800
1000
1200
1400
Temperature (°C)

Figure 4



R4
MicroVickers Hardness
0
100
200
300
400
500
600
700
800
900
1000
0
200
400
600
800
1000
1200
1400
Temperature (°C)

Figure 5

Alloy R4, which deviates from stoichiometry significantly, behaves quite differently in terms of hardness as a function of temperature. For this alloy, there is a small rise in hardness at the lowest temperatures, followed by a monotonic decrease with increasing temperature. The rate of softening is linear in two temperature regimes which correspond to the regimes for the other alloys, i.e., 400°C to 1000°C and above 1000°C. In the high temperature regime, the softening is considerably steeper than it is below 1000°C.

The two types of mechanical response observed in these alloys can be interpreted as indicative of the deformation mechanism. A regime of temperature dependence where softening is rapid, indicates thermally activated deformation and the presence of dislocation glide. On the contrary, shallow temperature dependence, such as observed in the near stoichiometry alloys at intermediate temperatures, indicates difficult dislocation motion and a good possibility for high temperature strength. These conclusions seem to apply quite well to the data at hand.

To further understand the deformation behavior, one must consider the role of defects. A number of interesting defect structures were observed by transmission electron microscopy (TEM) in these alloys after deformation at 1400°C, and these can be used to explain the microhardness results. The deformation defect structure of the far aluminum rich alloy, R4, was dominated by dislocations. These dislocations were observed to be free, that is, not pinned by other defects. The unrestricted mobility of the dislocations gives rise to the softening which is seen in the intermediate temperature region for this alloy. At high temperature, thermal activation, and possibly the activation of additional slip modes, further enhances dislocation plasticity, and softening occurs at a higher rate.

The dislocations which were observed in alloy R3 after deformation were in close association with a high density of planar sequence faults. The TEM study yielded experimental evidence for an attractive interaction between the dislocations and the sequence faults. Since the planar defects are essentially immobile, this attraction effectively pins the dislocations, limiting plasticity and giving rise to the hardness plateau at intermediate temperatures. Above about 1000°C, there is sufficient thermal activation to overcome the pinning and for deformation to proceed more freely, thus the hardness is seen to fall rapidly with temperature.

Clearly several important features of alloys based on Nb_2Al have been identified and elucidated. The formation of the Nb_3Al_2 tetragonal phase is entirely unexpected and has important implications for the development of multiphase alloys and composites based on Nb_2Al. Furthermore, this work demonstrates that the established assumption that this and other sigma phases do not exhibit dislocation mobility needs to be reassessed. The high temperature deformation of the alloys in this study, as seen in both the microindentation behavior and the direct TEM observations, is far more complex than previously thought. Not only does the mobility of dislocations depend on both temperature and composition, but there is evidence of a pinning mechanism which is similarly complex.

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